APPENDIX 4C
WASTE ANALYSIS QUALITY ASSURANCE/QUALITY CONTROL PLAN

ATTACHMENT 4 APPENDIX C
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1 INTRODUCTION

The procedures and methodologies used to characterize Munition Management Device, Version 1 (MMD-1) test wastes will ensure proper treatment of wastes in the reactor vessels; safe handling and storage of MMD-1 wastes; and safe handling, treatment, or disposal of MMD-1 wastes shipped offsite. This appendix summarizes the quality assurance/quality control (QA/QC) for sampling and analysis of MMD-1 waste streams. Waste sampling and analysis will be conducted in accordance with this plan. The Small Burials Contractor (SBC) will sample and analyze MMD-1 liquid and solid wastes for chemical agent concentrations. A Utah-certified laboratory (for example, Mountain States Analytical, GP Environmental Services, Inc.) will analyze liquid and solid samples for other hazardous waste constituents and characteristics in accordance with *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* current edition.

Detailed information on waste characteristics, waste analysis, and waste characterization methodologies is in Section 4 of the MMD-1 Resource Conservation and Recovery Act (RCRA) Research, Development, and Demonstration (RD&D) permit application.

1.1 PURPOSE

The primary purpose of waste sampling and analysis is to ensure that wastes are properly characterized in compliance with RCRA requirements for general waste analysis (40 CFR 264.13; R315-8-2.4). Waste sampling and analysis also ensures the safe management of wastes being stored or treated, proper disposition of treatment residues, and proper characterization of all process wastes for shipment offsite to an approved hazardous waste treatment, storage, and disposal facility (TSDF).

The plan presents requirements that will be followed to ensure waste sampling and analysis objectives are met and that all data obtained are technically sound, statistically valid, and properly documented. This plan also identifies the tools that will measure the degree of certainty that all objectives have been met.

1.2 QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES

Quality Assurance (QA) is a systematic approach to ensure that processes and activities meet quality, safety, technical, and management requirements, and that the data and results compiled for waste analysis are valid and properly documented. QA for the MMD-1 test will ensure that sampling, laboratory analysis, and air monitoring operations are performed in accordance with approved plans and procedures. Quality Control (QC) is the mechanism through which QA achieves its goals. The primary objective of this plan is to control and characterize any errors associated with the collected data. QA activities, such as using standard procedures for locating sample sites and collecting samples, are intended to limit the introduction of errors. QC activities, such as collecting duplicate samples and including blanks in sample sets, are intended to provide the information required to characterize any errors in the data. Other QC activities, such as planning the QC program and auditing ongoing and completed activities, ensure that the specified procedures are followed and that the QA information needed for characterizing errors is obtained.

The second QA/QC objective is to confirm that waste sampling and analysis have been conducted according to the specifications of the MMD-1 Waste Analysis Plan and requirements of this plan. QA/QC activities will include:

- Field inspections: performed by the QA Officer or designee, depending on the activity. The inspections will be primarily visual examinations but may include measurements of materials and equipment used, techniques employed, and the final products. The purpose of these inspections is to verify that a specific guideline, specification, or procedure for the activity is successfully completed.

- Field testing: performed onsite by the QA Officer or designee according to specified procedures.

- Laboratory analyses: performed on samples of waste by the onsite SBC laboratory for chemical agent or industrial chemical analyses or a contract laboratory for RCRA hazardous constituents/characteristics. Laboratory analyses will determine constituents or characteristics present and concentration level.

- Completing checklists: required for critical inspections. Checklists are to be filled out during the course of inspections to document the inspection and inspection results.

- Verifying instrument calibration: calibration records are maintained by both the SBC and contract laboratories on instruments used to perform waste sampling and laboratory analyses.

1.3 RESPONSIBILITIES AND AUTHORITY

The Program Manager for Chemical Demilitarization (PMCD) has been given direct responsibility for non-stockpile chemical materiel (NSCM); however, additional agencies are involved to varying extents. **Figure 4C-1** illustrates the various laboratory organization relationships that will be employed during the MMD-1 test activities. There will be a Project Officer from the PMCD Environmental and Monitoring Office assigned to monitor the SBC=s data collection activities for consistency and evaluate their quality control program. Dugway Proving Ground (DPG) will also provide oversight of laboratory activities and may conduct audits and/or surveillance of laboratory operations. The SBC will provide quality assurance personnel to document and ensure the integrity of all analytical data collected and obtained both from the onsite SBC laboratory and from the offsite contract laboratory.

The Project Manager for Chemical Non-Stockpile Disposal (PMCNSD) will be responsible for ensuring that appropriate data are provided to the state and Federal regulatory agencies, as stipulated by the RCRA RD&D permit. Monitoring staff will be assigned to monitor the SBC=s data gathering activities and to ensure compliance with environmental requirements. External audits and surveillances, either announced or unannounced, will be conducted as required. Additional external audits or surveillances will be conducted by other qualified organizations as requested by the Project Manager for Non-Stockpile Chemical Materiel (PMNSCM). All documents and data produced by the laboratories will be eligible for inspection. The environmental regulatory agencies may also review these data to ensure that MMD-1 test activity personnel are complying with permit requirements as they pertain to waste characterization and treatment operations. Onsite inspections may also be conducted by the U.S. Environmental Protection Agency (USEPA), Department of Health and Human Services (DHHS), and the Utah Division of Solid and Hazardous Waste. The objective of these inspections will be to make independent checks of the MMD-1 operating performance, including laboratory operations.

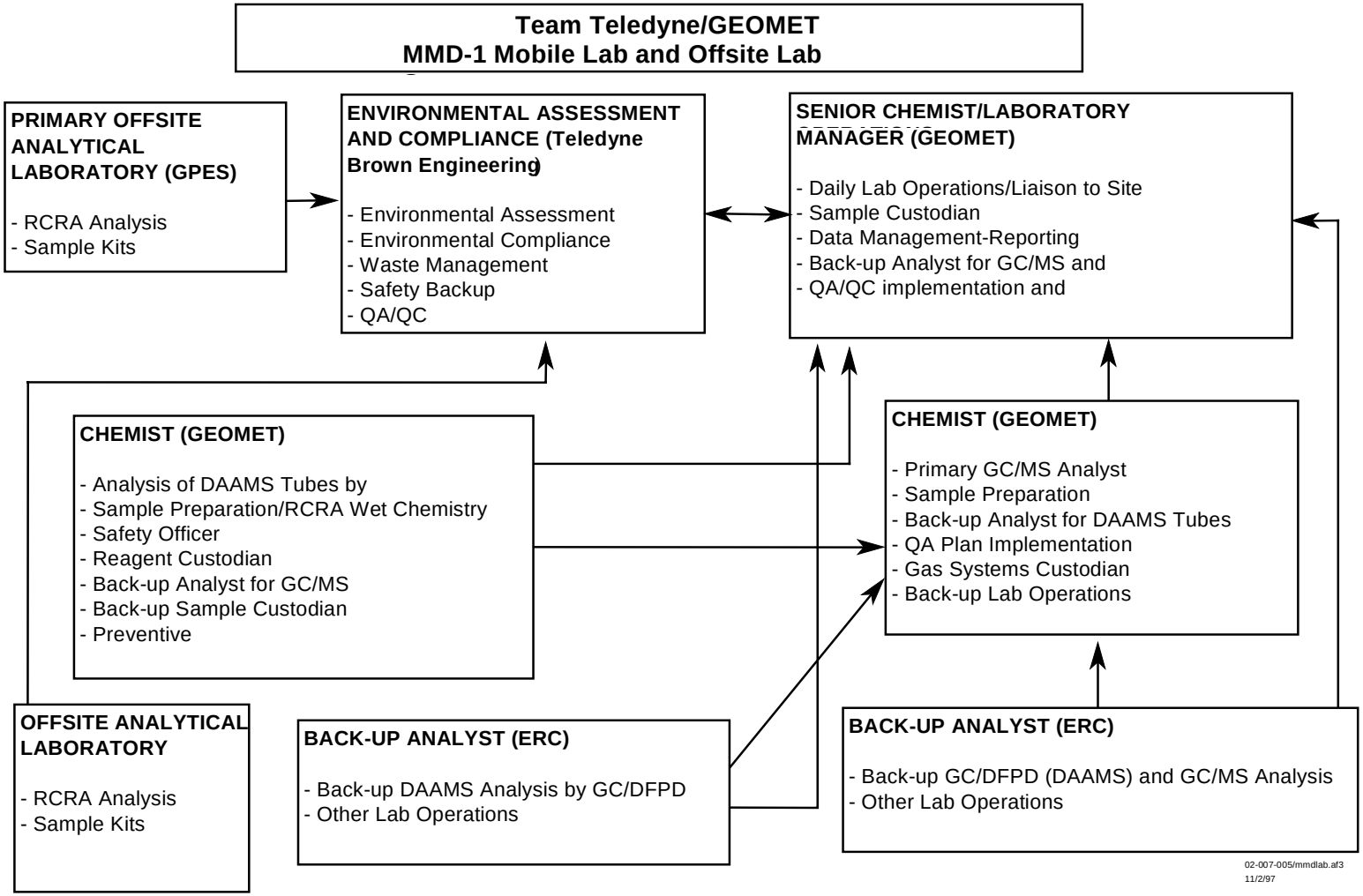

Figure 4C-1. Laboratory Organization Structure and Relationship

2 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) are qualitative and quantitative statements developed by data users to specify the quality of data needed from a particular activity. The USEPA provides the basis for developing the DQOs (USEPA, 1987).

The DQOs are the standards against which the data generated by a sampling effort can be evaluated. The DQOs for waste sampling and associated data analyses include, but are not limited to:

- Determining if waste samples are representative of the wastes at the time the samples were taken.
- Determining if the wastes selected for use in the MMD-1 test are within the limitations stated in the RCRA R&D permit (type and quantity).
- Determining if treatment residue (neutralent wastes) concentration values are within the treatment performance level goals.
- Sufficiently characterizing wastes that will be sent offsite to an approved hazardous waste TSDF.
- Ensuring laboratory analytical results can be validated.

2.1 DATA COLLECTION/SAMPLING OBJECTIVES

Collected data must be scientifically sound, of known quality, and thoroughly documented. The DQOs for data assessment are:

- Accuracy: the accuracy of an analytical method is represented as the mean of the percent recovery of the target analyte from a given matrix. The quality of the data can be assessed through the comparison of individual data values, expressed as percent recovery, to established QC limits. QC limits are derived from analysis of standard matrix QC samples, solvent (nonmatrix), QC samples, or surrogate analytes in field samples.
- Precision: the precision will be the agreement between the collected samples (duplicates) for the same parameters, at the same location, and from the same collection device.
- Representativeness: the representativeness will address the degree to which the data accurately and precisely represent a real characterization of the population, parameter variation at a sampling point, sampling conditions, and the environmental condition at the time of sampling. The issue of representativeness shall be addressed for the following points:
  - Based on the waste stream and its volume, an adequate number of samples are collected.
  - The representativeness of selected media has been accurately defined
  - The sampling and analytical methodologies are appropriate
  - The environmental conditions at the time of sampling are documented.

- Completeness: the completeness shall be defined as the ability of the sampling and analytical methodologies to accurately measure the constituents of concern present in the waste. The goal for completeness is 95 percent.

- Comparability: the comparability of the data generated shall be defined as the data that are gathered using standardized sampling methods, standardized analysis methods, and quality-controlled data reduction and validation methods.

3 SAMPLING QUALITY ASSURANCE/QUALITY CONTROL

Methods used to obtain a representative sample will be consistent with the sampling approaches and protocols described in Chapter Nine of *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition. The selected sampling methods used to characterize MMD-1 waste streams are summarized in Section 4 of the MMD-1 RCRA RD&D permit application, and are from: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, current edition, *Annual Book of ASTM Standards*, American Society for Testing and Materials (ASTM), current edition; or other USEPA-recognized methods. Air monitoring samples will be collected using the following Army methods: Munitions Management Device Version 1 MINICAMS[7] Standing Operating Procedures, and Dugway Proving Ground Methods selected from methods CL-001 through CL-051.

3.1 SAMPLE CONTAINER, PRESERVATION, HANDLING, AND MANAGEMENT

Sampling control procedures will be in place to ensure that each sample is accounted for at all times. The primary objectives of sample control procedures are:

- Each sample collected for analysis is uniquely identified

- Important and necessary sample constituents are preserved

- Samples are protected from loss, damage, or tampering

- Any alteration of sample during collection or shipping (e.g., preservation, breakage) is documented

- A record of sample custody and integrity is established that will satisfy legal and technical scrutiny

- The correct samples are analyzed and are traceable to the applicable data records (for example, chain-of-custody, field records, laboratory ledgers).

To ensure that representative samples of the MMD-1 waste streams are collected, samples must be collected in certified clean sample containers, properly preserved, and transported to the laboratory to minimize significant changes in constituents. Certificates of Analysis for the sample containers will be retained by the MMD-1 Project Environmental Scientist.

Sample containers used for waste analysis will arrive from the contract laboratory prepared with preservatives as appropriate for the parameter or analyte of interest. Containers with preservatives will be labeled or marked as containing preservatives. If wipe sampling is conducted for closure activities, wipe

sampling materials will be moistened with the appropriate wetting substance (for example, solvent) prior to sampling.

The MMD-1 QA staff will monitor the receipt of samples and compliance with preservation and holding time specifications. This includes checking the pH of preserved samples before sealing the sample containers for transport to the laboratory. Field pH verification will be performed using pH paper (gross check). However, no samples collected for volatile organics analysis (VOA) will be field checked for pH. The receiving laboratory will be responsible for verifying the pH of samples for VOA, indicating the sample was properly preserved at the time of collection. Once samples for VOA are sealed, the sample containers should not be opened until ready for analysis. A copy of the sample log sheet will always accompany the samples as part of the chain-of-custody (COC) record.

As part of sample management procedures, personnel collecting the samples will maintain a record of sampling activities. Sampling records typically include: the purpose of sampling; date and time of collection; sample number; sampling location, sampling methodology, container description, description of sample preservative, pH verification, and waste description (ash, salt, brine, etc.); description of process originating the waste; name and address of field contact; number and volume of samples; field observations; destination and transporter; and signature of collector.

As applicable, equipment used to sample waste materials will be the disposable type or designed for easy decontamination. Contaminated disposable-type equipment will be managed as hazardous waste, as appropriate. Cleanable equipment will be thoroughly decontaminated prior to reuse. Decontamination solutions will be managed as hazardous waste, as appropriate.

All samples will be collected, contained, preserved, transported, received, prepared, and analyzed in accordance with USEPA-published guidance (including SW-846, current edition), DOT, and Army requirements.

3.2 CHAIN-OF-CUSTODY

COC documentation as described by the EPA National Enforcement Investigation Center (NEIC) will be strictly adhered to and will be used to track samples through the SBC onsite laboratory, DPG laboratory, and the offsite contract laboratory. Evidence of sample custody shall be traceable from the time the samples are collected, delivered to a laboratory, analyzed, and disposed of after sample analysis. After samples are collected, the COC form will be completed, and the original and one copy will be placed in a plastic bag inside the secured sample transport container. A custody seal will be affixed to each sample container. One copy of the COC form will be retained onsite, and the original COC form will be forwarded to the analytical support laboratory data coordinator after samples are received in the laboratory. A copy of the COC form will be forwarded with the samples as they are received in the laboratory. A copy of the COC form will be forwarded with the samples as they are stored, prepared, analyzed, and finally disposed of in the laboratory. A sample receipt form will be faxed to the SBC MMD-1 Project Environmental Scientist immediately upon arrival of shipped samples, noting any sample breakage or other damage that may have occurred. Any problems with sample breakage, missing samples, or shipping logistics will be documented and resolved between the SBC MMD-1 Project Environmental Scientist and the offsite and/or onsite Analytical Laboratory Manager (or designated personnel). Receipts from post offices, copies of bills of lading, and airbills will be retained as part of the COC documentation. An example of a COC form is shown in **Figure 4C-2.**

3.3 FIELD SAMPLING AND LABORATORY QUALITY CONTROL

The Quality Control requirements for the process waste streams generated from the MMD-1 test activities can be separated into two categories: Quality Control for agent screening and Quality Control for RCRA waste characterization. The SBC onsite Mobile Laboratory will perform the agent screening according to PMNSCM approved methods. The offsite laboratory will perform the RCRA characterization according to EPA SW-846 protocols.

Principal elements of the sampling and field QA/QC strategy include:

- Developing sound sampling approach based upon the intended use of the data
- Using sampling methodologies which allow the collection of representative samples based upon data needs
- Using sampling devices that minimize the disturbance or alteration to the media=s chemical composition
- Using disposable sampling devices to eliminate potential cross-contamination between sample points
- Employing decontamination procedures which reduce cross-contamination potential between sampling points
- Using proper sample containers and preservation techniques that maximize the integrity of the samples.

All field control samples shall be handled exactly as the environmental samples. The identity of all field control samples collected must be held blind to the laboratory until the data are reported.

CHAIN-OF-CUSTODY RECORD
Generator Name, Address
Project No./ID _____ Sheet 1 of _____
Sampled By: _____ Bottles Supplied By: _____
NOTE: When samples are received, return this form to:
Name: _____ at the above address.

Sample I.D.	Sample Description/Location	Collection Date/Time	Number of Bottles	Analyses Requested

Sample Received and Packed by: _____ Date/Time: _____
Cooler/Box Identifier: _____ Method of Shipment: _____
Date of Shipment: _____ Airbill No. _____
Sample Disposition: _____ Return to Generator
_____ Dispose of by Laboratory
$_____ Disposal Fee

Laboratory Name: _____
Received by: _____ Date/Time: _____
Seals Intact (Y/N): _____ Containers Intact (Y/N): _____
If not intact, please describe in Comments section.

NOTE: Laboratory's chain-of-custody procedures are in effect from receipt through analysis.

SAMPLES RELINQUISHED BY:			SAMPLES RECEIVED BY:			SAMPLES INTACT
Signature	Date	Time	Signature	Date	Time	

Comments: _____

_____

Figure 4C-2. Example of a Chain-of-Custody Record

3.3.1 Field Quality Assurance Quality Control for Agent Sampling and Analysis

Table 4C-1 lists the types of samples that will be collected and analyzed for the presence of chemical agent during the MMD-1 test. The number and frequency of Quality Control samples collected and analyzed for agent screening will be higher than those collected for RCRA waste characterization to satisfy the need for strict validation of the sampling and analysis activities for chemical agent.

Table 4C-1. Field QC Samples for Agent Analysis[a]

Waste Stream	Field Duplicate[b]	Laboratory Duplicate[c]	Equipment Blank
Liquid[d] (Neutralent)			
MTV	1	1	NA
LRV	1	1	NA
Surge Tanks	1	1	NA
Liquid[d] (Process Wastes)			
Rinsates	1	1	1
Canned Pump Reservoir Fluids (Barrier Fluid)	1	1	1
Cooling Water	1	1	1
UPA Waste	1	1	1

NOTES:

a The frequency of sampling events for each waste stream is presented in **Table 4-10** of Section 4 of this MMD-1 RCRA permit application. Parameters for process waste evaluation, waste characterization methods, and equipment and sampling methods will be per **Tables 4-7**, **4-8**, and **4-10** in Section 4 of the MMD-1 RD&D permit application.

b A field duplicate will be analyzed per campaign or 20%, whichever is greater.

c A sufficient sample volume or mass will be collected at each sampling point for each analysis such that a laboratory duplicate may be prepared and analyzed.

d Minimum sample requirements for liquid samples will be per **Table 4-11** in Section 4 of the MMD-1 RD&D permit application.

LRV = Liquid Reactor Vessel
MTV = Munition Treatment Vessel
N/A = not applicable
UPA = unpack area

3.3.2 Description of Field and Laboratory QC Samples

Table 4C-1 illustrates the types and frequency of QC samples required to satisfy the elements of a sound sampling strategy for agent screening. The current sampling device for liquid waste samples is a 150 mL sample bomb. For each liquid sampling point [that is, Liquid Reactor Vessel (LRV), surge tanks, etc.], enough sample will be collected in the sample bomb to allow for preparation of a duplicate sample aliquot for analysis in the laboratory. All liquid matrices will be analyzed in duplicate. For solid samples, duplicate aliquots will be prepared in the field (if collected for laboratory analysis). Field blanks shall be prepared at a frequency of 1 per 10 samples. The field blanks for liquid matrices will be collected and analyzed as a substitute for trip blanks.

Equipment blanks (or rinsates) will be collected from rinsates of sampling equipment after decontamination to verify that the sampling equipment is properly decontaminated. The sample bombs used for liquid sample collection will be cleaned and treated as field blanks. No field spike samples will be prepared for agent screening; therefore, matrix spike and matrix spike duplicate analysis will be used to evaluate target analyte recovery in various process waste matrices.

For laboratory prepared QC samples, a typical extraction batch and analytical sequence should include the following but not be limited to:

- Method Blank (with solvent lot number identifier)
- Laboratory Control Sample
- Original Sample (OS)
- Original Sample Duplicate (OSD)
- Original Sample Matrix Spike (MS)
- OS Matrix Spike Duplicate (MSD)
- OS 2
- OS 2 Duplicate (OS2D)
- Additional samples as necessary, up to 10 analytical samples
- Continuing Calibration Verification (CCV).

3.3.3 Field Quality Assurance/Quality Control for RCRA Waste Sampling and Analysis

The goal of the RCRA waste sampling and analysis effort is to provide representative information regarding the characteristics of the process wastes generated during the MMD-1 test activities. The offsite laboratories chosen for this RCRA analysis will be Utah-State certified, have ignificant capacity and quality to meet analytical demand, provide timely and complete data pacakages, and provide technical support in the form of sample kits and shipping supplies. For a project of this scope, a primary offsite laboratory and a back-up laboratory may be chosen. The frequency and type of field Quality Control samples required to support validation of the data generated will be less stringent than those required for the agent screening, but will provide sufficient statistical information to evaluate the waste streams prior to shipment offsite to a TSDF. An initial approach to the field Quality Control strategy for RCRA waste is detailed in **Table 4C-2.**

3.3.4 Description of Field and Laboratory QC for RCRA Characterization of Waste Streams

Table 4C-2 illustrates the type and frequency of QC samples required to satisfy the elements of a sound sampling strategy for RCRA characterization of process wastes. A field blank sample will be prepared in the laboratory and transported onsite with sample containers. The field blank sample matrix must be representative of the sample being collected. The field blank container will be opened during the sample event for the sample duration it takes to collect one sample. The field blank sample will be prepared at a frequency of 1 per 10 analytical samples. Each group of constituents [that is, Toxicity Characteristic (TC) metals, TC organics] will have an accompanying field blank. The field blank will be used as a substitute for a trip blank and will accompany the samples to the offsite RCRA laboratory. The field blanks will be treated as discrete analytical samples and are used to verify elimination of cross-contamination during sampling and transport. One sample point (for example, surge tank) will be randomly chosen as the QC sample, and a field duplicate and matrix shall be prepared from that waste stream. Enough sample in the proper sample container with the appropriate preservative shall be collected to allow the analytical laboratory to meet method requirements. Field duplicate and matrix spike samples shall be collected at a frequency of 1 per 10 process waste samples.

Table 4C-2. Field QC Samples for RCRA Waste Characterization[a]

Waste Stream	Field Duplicate[b]	Field Split Samples[c]	Laboratory Duplicate[d]	Equipment Blank	Trip Blank[e]/ Field Blank
Liquid[f] (Neutralent)					
Surge Tanks	1	1	1	NA	1
Liquid[f] (Process Wastes)					
Rinsates	1	1	1	1	1
Canned Pump Reservoir Fluids/Barrier Fluid		1	1	1	1
Cooling Water	1	1	1	1	1
UPA Waste	1	1	1	1	1
Solids[g]					
Solids from MTV Strainers	1	1	1	1	1
Plastic, Soil, Debris Rags, Paint Chips, Bead Blast Residue, Gaskets, Swatches, Dunnage - After Treatment	1	1	1	1	1
Overpack Material	1	1	1	1	1

NOTES:

a The frequency of sampling events for each waste stream is presented in **Table 4-10**. Parameters for process waste evaluation, waste characterization methods, and equipment and sampling methods will be per **Tables 4-7**, **4-8**, and **4-10** in Section 4 of the MMD-1 RD&D permit application.

b One field duplicate will be analyzed per surge tank generated. For other wastes field duplicates will be collected at 20 % of the total number of waste containers generated per waste stream for each campaign, whichever is greater.

c One field split sample will be collected per sampling event for each waste stream. The split sample will be sent to the offsite laboratory for RCRA analyses.

d A sufficient sample volume or mass will be collected at each sampling point for each analysis such that a laboratory duplicate may be prepared and analyzed. The frequency of lab duplicates is one per analytical batch or 20%, whichever is greater.

e Volatile organics only.

f Typical sample requirements for liquid samples will be per **Table 4-11** in Section 4 of the MMD-1 RD&D permit application.

g Typical sample requirements for solid samples will be per **Table 4-12** in Section 4 of the MMD-1 RD&D permit application

For laboratory prepared QC samples, a typical extraction batch and analytical sequence will follow the SW-846 method applied (or other approved method) exactly.

3.3.5 Definitions

Analytical Batch- A batch is defined as the number of samples of similar matrix that are processed as a set through the entire preparation and analytical process. The maximum size of an analytical batch for this project is 20 samples.

Method Blank- A method blank is an aliquot of reagent water or a representative solid matrix for soil/solid samples that is carried through the entire analytical procedure for each analytical batch. Method blanks will be used to determine the existence and magnitude of possible contamination encountered during the entire sample preparation and analysis process. The concentration of target analytes in the method blanks should be below the Instrument Detection Limit.

Laboratory Control Samples- A laboratory control sample (LCS) consists of a certified reference material spiked with analytes representative of the target analytes. LCS=s are used to verify that bias of the analytical process are within control limits specified in the method Standing Operating Procedures (SOP). The Matrix of the LCS=s is comparable to the matrix samples analyzed in the analytical batch.

Laboratory Surrogates and Internal Standards- A surrogate standard consists of spiking samples and blanks with known concentrations of certified non-target analytes before analysis of samples. The recovery acceptance criteria of surrogate standards are specified in the method SOPs.

Laboratory Matrix Spike- A matrix spike is an aliquot of a sample spiked with known quantities of analytes and subjected to the entire analytical process. It is used as a measure of the percent recovery of analytes in the matrix of interest.

Field Duplicate Samples- Field duplicate samples are independent field samples collected as close as possible to the same point in space and time. Field duplicates are used to confirm precision of analytical data.

- $ Using sampling methodologies which allow the collection of representative samples based upon data needs
- ● Using sampling devices that minimize the disturbance or alteration to the media=s chemical composition
- $ Using disposable sampling devices to eliminate potential cross-contamination between sample points
- $ Employing decontamination procedures which reduce cross-contamination potential between sampling points
- ● Using proper sample containers and preservation techniques that maximize the integrity of the samples

All field control samples shall be handled exactly as the environmental samples. The identity of all field control samples collected must be held blind to the laboratory until the data are reported.

3.4 HEALTH AND SAFETY PROTOCOLS

During all sampling activities, strict compliance with industrial hygiene and safety standards will be mandatory. All personnel involved in sampling activities will be trained in the applicable safety procedures; the use of all cleaning, decontamination, and sampling equipment; and proper cleaning and decontamination techniques. Sampling personnel will have received Occupational Safety and Health Administration (OSHA) health and safety training for hazardous waste operations. Sampling personnel will be required to wear eye, skin, and respiratory protection gear, as dictated by MMD-1 project safety personnel. If personnel accidentally contact waste material, decontamination procedures will be performed as directed by MMD-1 project safety personnel in accordance with Army requirements.

Waste samples will not be collected and sent for analysis unless the samples meet the following guidelines prior to releasing them to the onsite SBC laboratory or to the offsite laboratory for further analysis/waste characterization. Sample released to the onsite SBC laboratory should not contain chemical agent or industrial chemicals in concentrations or amounts above the values defined for research, development, test and evaluation solutions in Army Regulation 50-6, Table 9-1 (see **Table 4C-3**). Samples released to the offsite laboratory should not contain chemical agent in concentrations above the treatment level of 50 mg/L.

4 REFERENCE MATERIALS AND CALIBRATION STANDARDS

4.1 INTRODUCTION

The standard solutions used for method or monitor calibration, along with the reference materials from which they are made, play an important role in QC because no chemical analysis method can be more accurate or more reliable than the standard solutions that are used for method calibration.

The standards used for QC samples and calibrations will be prepared at the highest purity available.

The chemical agent calibration solutions will be issued by the SBC in dilute standards and will be traceable to the original Chemical Agent Standard Analytical Reference Material (CASARM) that will be provided by the Edgewood Research, Development and Engineering Center (ERDEC).

4.2 REFERENCE MATERIAL STANDARDS PROGRAM

Reference standards are required to calibrate and challenge instruments and spike QC samples. These solutions must be of known concentration and purity to validate analytical results. Standards used to conduct analytical analyses will be either standard reference materials (SRMs) or CASARMs where possible. SRMs are developed and distributed by the National Institute of Standards and Technology (NIST). CASARMs are chemical agent reference materiel that are of characterized composition and purity, and are developed and distributed by ERDEC. DPG Chemical Test Division will provide calibration standards for the SBC Mobile Chemical Laboratory to support the MMD-1 monitoring operations and will not exceed RDTE concentrations of agent in solution.

All calibration solutions and standards to be used during the MMD-1 test will be prepared and maintained under a laboratory standards tracking system. This system will ensure that preparation, checking, documentation, storage, and disposal of standards will be performed in accordance with specified procedures and schedules that are appropriate for each analyte of interest.

Table 4C-3. RDT&E Solution Levels

Chemical Agents	Level
HD	Concentrations not greater than 10 mg/mL (chemical agent/solvent) and containing no more than 100 mg of chemical agent
GB	Concentrations not greater than 2 mg/mL (chemical agent/solvent) and containing a maximum quantity of 20 mg of chemical agent
VX	Concentrations not greater than 1 mg/mL (chemical agent/solvent) and containing a maximum quantity of 10 mg of chemical agent

5 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

An analytical laboratory must conduct its operations in such a way as to provide reliable information. The QA/QC of data generated by the SBC onsite analytical laboratory and offsite Utah-certified laboratory that will perform RCRA analyses will be controlled by a laboratory Quality Assurance Plan. The QA Plan at a minimum will document the following:

- Sample custody and management practices
- Sample preparation and analytical procedures
- Instrument maintenance and calibration procedures
- Internal QA/QC measures including the use of method blanks where appropriate, checklists, known QC check sample and laboratory duplicate samples.

The offsite analytical laboratory will submit a Statement of Qualifications and a Quality Assurance Manual to be retained by the SBC Laboratory Manager.

5.1 PREVENTIVE MAINTENANCE

Preventive maintenance will be conducted to ensure that the sampling and analysis equipment is in the proper operating condition, and will prevent problems before they affect the validity of the data. The hardware associated with sampling and analysis will be maintained in accordance with the vendor or manufacturer=s recommendations. The preventive maintenance activities will be documented and supported by a preventive maintenance schedule.

Preventive maintenance of field-monitoring equipment will be performed by the personnel responsible for operating the equipment in accordance with the equipment operations and maintenance (O&M) manual or the vendor=s recommendations. If an outside vendor is contracted for maintenance activities, the equipment operator will be responsible for ensuring all maintenance is scheduled and performed in accordance with manufacturer recommendations and operator=s experience.

For analytical equipment located in the analytical support laboratories, preventive maintenance will be performed in accordance with the manufacturer=s recommendation and on an as-needed basis as deemed necessary by the instrument operator. The operator may choose to perform preventive maintenance when the performance of the instrument begins to show a slight degradation from its usual analytical performance. Possible causes of performance degradation may be due to the lack of peak resolution, poor analyte recovery on a QC sample, or a shift in retention times.

The instrument, including manufacturer, model, and accessories, will be specified in the preventive maintenance records. Preventive maintenance will be performed by qualified personnel. Records of preventive maintenance, repairs, adjustments, and calibrations will be maintained in a log book and made available for inspection upon request.

A preventive maintenance schedule will be documented for all monitoring and analytical equipment. The schedule will include the name of the individual(s) responsible for performing the preventive maintenance.

5.2 ROUTINE ASSESSMENT OF PRECISION, ACCURACY, AND COMPARABILITY OF ANALYTICAL DATA

Quality assurance for analytical data from collected samples will include evaluation of precision, accuracy, and comparability. These are discussed in the following paragraphs.

5.2.1 Precision

Precision is a measure of agreement among different analyses performed using the same test method. It is estimated by determining the standard deviation between duplicate samples. One field duplicate sample will be collected per sampling event for each waste stream. Split samples will be taken for each sampling point for all waste streams. (See **Table 4C-2**.) Laboratory precision is only one part of the total process precision measurement, from sample collecting to data reporting. Selecting an appropriate precision level should not be based on what is attainable in the analytical support laboratory. Once the sample has been received in the laboratory, most of the sample-to-sample variation has already been introduced into the sample measurement process.

Analyzing field and laboratory duplicates will provide information on the precision of the analyses of the analyte and the sample matrix. Precision will be assessed by analyzing field and/or matrix spike duplicates, calculating the relative standard deviation (RSD), and comparing the RSD to the DQO. An additional measure of precision that will be used is control charting of derived information from duplicate analyses.

5.2.2 Accuracy

Accuracy relates the amount of an element or compound recovered by the analytical procedure to the amount of analyte present. The accuracy of measurement for the samples sent to the laboratory for analysis is controlled primarily by the laboratory and is reported as percent recovery. For results to be accurate, the analysis must yield found values close to the true value. Calibration curves, spiked samples, and calibration checks all assess and control the accuracy of the laboratory results, as well as the comparability of the results. In addition to the sample matrix spikes, QC check samples, consisting of spikes into analyte-free water (or extraction solvent), will be analyzed. The measured values for the spiked solutions will be compared with the target value to assess the spiking and extraction efficiency and accuracy. The extraction efficiency will be determined for different analytes and matrices that require extraction methods. If the percent recovery for actual field sample spikes is less than 75 percent or greater than 125 percent while spiked water (or extraction solvent) shows acceptable recoveries, then strong evidence exists that the sample matrix is causing erroneously low or high recoveries and is affecting the accuracy of the method. Accuracy of laboratory analysis will be assessed based on calibration curves and percent recovery. An additional measure of accuracy that will be used is control charting of all derived recovery information from laboratory control samples and matrix spike samples.

5.2.3 Comparability

Comparability is the degree to which one data set can be compared with another. Comparability is achieved by using consistent methods and standards that are traceable to a reliable source.

Comparability can be enhanced by using:

- EPA SW-846 or EPA 600/4-88-039 methods of analysis
- ASTM methods
- National Institute for Occupational Safety and Health (NIOSH) methods
- American Association of Official Analytical Chemists methods
- Other methods that are standardized and approved by PMCD and reporting data in conventional and standard units.

5.3 DATA QUALITY OBJECTIVES AND DATA DELIVERABLES

5.3.1 Data Quality Objectives

The methods for agent screening are currently under development, and the program control limits for QC analyses have not been finalized yet. **Table 4C-4** presents proposed acceptance criteria for QC analyses including duplicates, quality plant (QP) samples, and the matrix spike/matrix spike duplicate for agent screening performed by the onsite Mobile Chemical Laboratory. Note that these values are proposed acceptance criteria for the analyses listed. The methods for agent analysis have not been field tested. Actual duplicate RPDs may vary according to matrix and agent. Surrogate compounds, if used in the approved methods, shall be tracked as a control indicator, and acceptance criteria shall be established. The offsite RCRA characterization will conform to the data quality objectives (DQOs) specified in the methods applied (i.e., EPA SW-846).

Table 4C-4. QC Data Acceptance Criteria For Agent Analyses

Acceptance Criteria for Agent Screening - Spike Recovery Analyses

	Acceptance Criteria[a]		
Analysis Method	**ICV/CCV (or QL for DAAMS analysis)**	**Laboratory Control Samples**	**Matrix Spike Samples (QP for DAAMS)**
GC/MS (liquid extracts)	85-115%	85-115%	75-125%
GC/FPD (DAAMS or MINICAMS[7])	85-115%	NA	75-125% (QP)

NOTE:

a Acceptance criteria for percent recovery of the target analyte from the true value. The methods for agent analysis have not been field tested. Actual recoveries may vary according to matrix and agent.

CCV = continuing calibration verification
DAAMS = Depot Area Air Monitoring System
FPD = flame photometric detector
ICV = initial calibration verification
QL = quality lab
QP = quality plant

Acceptance Criteria for Agent Screening - Duplicate Results

	Acceptance Criteria[b]		
Analysis Method	**Laboratory Control Samples**	**Matrix Spike Samples**	**Field Duplicates**
GC/MS (liquid extracts)	+/-20%	+/-20%	+/-20%

NOTE:
b Acceptance criteria for relative percent difference (RPD) of the duplicate analyses are listed. The methods for agent analysis have not been field tested. Actual duplicate RPDs may vary according to matrix and agent.

5.3.2 Data Quality Assessments and Data Deliverables

Data quality assessments will evaluate whether the data generated by the onsite and offsite laboratories is consistent with the established DQOs specified in the Quality Assurance Project Plan for the MMD-1 test at DPG.

For the onsite Mobile Chemical Laboratory, the laboratory technicians will be responsible for reviewing all raw data generated during agent screening on a daily basis. In general, data packages generated by one analyst will be reviewed by a different analyst to ensure thorough review. A signature of the reviewer in ink with the review date shall be written on the data package cover sheet to document the review process. The laboratory supervisor will be responsible for reviewing all processed and raw data and supplying the specified deliverables to the project QA Coordinator. The MMD-1 QA Coordinator or assignee shall begin data quality assessment procedures with the validation of the equations and calculations used for calibration curve evaluation, percent recoveries of spiked samples, duplicate relative percent differences (RPDs), QC sample results, and samples analysis.

For the RCRA waste characterization, the offsite laboratory will furnish a Quality Assurance Manual (or Quality Assurance Program Plan) that defines the quality procedures and policies specific to that facility. The SBC Environmental Scientist, Laboratory Manager, and QA Coordinator will be responsible for reviewing the reports from that facility to ensure consistency with RCRA limits and with the furnished QA Manual. An audit of the analytical laboratory may be performed by the project Environmental Scientist or assignee at any time during the MMD-1 test.

5.3.2.1 Report Deliverables for Agent Screening

Report deliverables for agent screening for the MMD-1 test will include routine summary reports for daily submittal. All raw data will be archived in a consistent manner facilitating retrieval for the closure report or during audits. The daily summary reports for method blanks, analytical samples, and QC samples will include the following information:

- Chain of Custody, Field Sampling Logs, any associated correspondence
- Name and address of laboratory (on letterhead)
- PMNSCM or other approved method used (with title and method number)
- Client delivery order (or job) number
- Sample identification, client, and laboratory number
- Date and time sampled
- Date and time sample received by laboratory
- Date and time sample was extracted/digested
- Dilution factor
- Sample matrix
- Date and time sample was analyzed
- Parameters tested
- Units reported
- Concentration of each parameter found
- Reporting limit or other similar limit for each parameter [practical quantitation limit (PQL)]
- Report date
- Signature of laboratory supervisor or director (or assignee).

All data packages will have the following additional information:

<u>GC/MS</u>

- Sample table
- Tune report
- Calibration report
- Continuing calibration report
- Extraction log(s)
- Method blank results
- Lab control sample (% recovery)
- Matrix spike (% recovery)
- Surrogates (% recovery).

DAAMS Analysis

- Sample information page
- Extraction/tube preparation log
- Calibration report
- Continuing calibration report
- Method blank results
- Spiked sample results (% recovery).

Additional QC Results

Additional QC summary reports may be delivered to the MMD-1 QA Coordinator and could include such information as field duplicate analysis results (% RPD) and control charts for duplicate sample analysis and control charts for spike recovery samples. A narrative may also be delivered which would include:

- A description of any modifications used to analyze samples
- Description of any unusual situations or problems encountered during transport or analysis of samples and a summary of any corrective actions taken. If no problems were encountered, then a statement to that effect should be written
- A list of QC samples
- An explanation of terminology, acronyms, and qualifiers used in the report
- Signature of laboratory supervisor, director, or assignee.

5.3.2.2 RCRA Characterization Deliverables

For the offsite RCRA laboratory, data packages will consist of complete data packages with raw data. A request for electronic deliverables will be made. All summary reports for the RCRA characterization should include:

- Executive Summary
- Chain of Custody, any associated correspondence
- Name and address of laboratory (on letterhead)
- EPA or other approved method used (with title and method number)
- Client delivery order (or job) number
- Sample identification, client, and laboratory number
- Date and time sampled
- Date and time sample received by laboratory
- Date and time sample was extracted/digested
- Dilution factor
- Sample matrix
- Date and time sample was analyzed
- Parameters tested
- Units reported
- Concentration of each parameter found
- Reporting limit or other similar limit for each parameter (PQL)
- Report date

- Signature of laboratory supervisor or director (or assignee)
- QC Associate
- Raw Data as requested

5.3.2.3 Quality Control Deliverables for RCRA Characterization

Metals - Toxicity Characteristic (TC) extraction logs (EPA 1311), method blank results, lab control sample and lab control sample duplicate (% recoveries with calculated RPD), matrix spike and matrix spike duplicate (% recoveries with calculated RPD), OS and OSD with calculated RPD.

Organics - extraction logs, method blank results, lab control sample, (% recovery), and matrix spike (% recovery), and surrogates (% recovery).

Wet Chem - extraction preparation logs, method blank results, lab control sample and lab control sample duplicate (% recoveries with calculated RPD), matrix spike and matrix spike duplicate (% recoveries with calculated RPD), OS and OSD with calculated RPD.

Other - For methods in which no spikes can be performed (that is, specific gravity, flashpoint) and original sample and original sample duplicate analysis must be performed and reported (OS/OSD with calculated RPD%).

5.3.3 Data Qualifiers

In cases where results are out-of-control and the laboratory supervisor and the QA Coordinator determine that re-preparation and re-analysis is not possible, then the results for that sample or analytical batch shall be qualified. The qualifiers used for the MMD-1 test shall be consistent with the USEPA Contract Laboratory Program data qualifiers that are listed and defined below.
Additional notes and explanations may accompany the reports to further describe the occurrence.

ND: This flag indicates the compound was not detected at or above the MDL.

U: This flag indicates the compound was detected above the MDL but below the PQL .

J: This flag indicates an estimated value. This flag is used (1) when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed and (2) when the mass spectral and retention time GC/MS identification criteria and the result is less than the Practical Quantitation Limit (PQL) but greater than zero.

N: This flag indicates presumptive evidence of a compound. This flag occurs only on a mass spectral library search. It is applied to all total ion chromatogram (TIC) results.

B: This flag is used when the analyte is found in the associated method blank as well as in the sample. It indicates probable blank contamination and warns the data user to take appropriate action. This flag is used for TICs as well as positively identified target compounds.

E: This flag indicates compounds with concentrations exceeding the upper level of the calibration range of the instrument for that analysis. If one or more of the compounds have a response greater than the upper level of the calibration range, the sample or extract shall be diluted and re-analyzed. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, both results are delivered with the flag ADL@ attached to the second analysis.

D: This flag is used for all compounds identified in an analysis at a secondary dilution factor.

X: Other specific flags may be required to properly define the results. If used, the flags shall be fully described, with the description attached to the sample summary package. Use Y and Z if more than one flag is needed.

R: This flag is used to indicate that the analytical result is rejected. A reason for the data rejection is required.

5.4 CORRECTIVE ACTION

Corrective action will be initiated by the laboratory QA/QC Officer when required to ensure data quality meets the criteria established in the laboratory QA Plan. Corrective action of QA activities will be initiated by the Laboratory Manager (SBC or offsite laboratory) when potential or existing conditions are identified that may adversely impact data quality. Events that will require corrective action include noncompliance of approved analytical procedures, noncompliance of approved standard operating procedures (SOPs), out-of-control conditions, absence of proper or approved procedures for a sampling or analytical activity, material nonconformances, and inability to attain DQOs.

The need for corrective action must immediately be documented and reported to the laboratory manager. The corrective action may be immediate or long-term. An immediate corrective action may be the recalculation of results, reanalysis of samples, or repeat of sample collection. A long-term corrective action may include an increase in QC samples, more frequent calibrations, implementation of control charts, or additional backup equipment.

Any necessary corrective measures identified by the offsite contract laboratory due to problems such as broken sample containers, COC not signed, analytical errors, etc. will be immediately made known to the SBC Laboratory Manager, SBC MMD-1 Project Environmental Scientist, and QA/QC Officer for resolution.

Standard laboratory-initiated corrective actions consist of checking instruments and apparatus, obtaining new reagents and/or standards, and calibration verification or recalibration. If standard laboratory-initiated corrective actions do not identify and correct the problem, then instrument custodians, instrument system specialists, method authors, or chemists will troubleshoot and repair or correct the system performance.

For either immediate or long-term corrective actions, steps comprising a closed loop corrective action system are as follows: (1) define the problem; (2) assign responsibility for investigating the problem; (3) investigate and determine the cause of the problem; (4) determine a corrective action to eliminate the problem; (5) assign and accept responsibility for implementing the corrective action; (6) implement the correction and determine its effectiveness; and (7) verify that the corrective action has eliminated the problem.

Undesirable performance or analytical errors will be identified as a deficiency in bias or accuracy. Either the results of replicate measurements were not in close agreement or the results were not in agreement with the expected (target) or reference values. Also, there is a possibility that both situations will occur concurrently. Rules for finding and resolving the causes of these deficiencies are not well established. However, the approach that the analyst takes must be systematic and based on common knowledge and experience of the laboratory personnel. A team effort involving the analysts, the immediate supervisor, and the Laboratory Manager will be required. The most obvious causes for deficiencies will be evaluated first. If the initial investigation does not resolve the problem, attention will be directed to the more complex possibilities.

Obvious errors, such as errors in transposition and transcription errors of data, using incorrect calculations or errors in calculations, incorrect readings and recording of instruments readouts, using the wrong analytical procedures, and the lack of attentiveness to details in the laboratory will lead to imprecision or bias. A review and internal audit of the data and a detailed discussion with the analysts concerning how and when they performed specific steps in the laboratory procedures may indicate the cause of, and a corrective action for, the deficiency.

Causes and recommended resolutions of bias and accuracy are discussed below.

Bias

Inexperience of the analyst, instrument instability, variable contamination in the samples, variability of the method blanks, poor reagent quality and control, or fluctuations of the laboratory environment are possible causes of bias. Approaches to resolving these causes are the following: (1) check for obvious errors first; (2) repeat the analysis at the point where the sample is first introduced into the analytical procedures; (3) repeat the analysis on a different instrument or use another GC column; and (4) have another analyst repeat the analysis.

Accuracy

Incorrect calibrations, losses of analyte during sample preparation or analysis, incorrect calibration standards, stock solutions, innate bias of the analyst, matrix effects on the analyte, instrumental shifts, instrument not calibrated, and contaminations in the sample or standards are possible causes of inaccuracy. Approaches to resolving these causes are the following: (1) check for obvious errors first; (2) repeat the analysis at the point where the sample is first introduced into the analytical procedure; (3) repeat the analysis with new calibration standards; (4) recalibrate the analytical instrument; (5) repeat the analysis on another instrument that is calibrated; (6) have another analyst repeat the analysis; (7) repeat the analysis with fresh or new samples, if possible; and (8) check analytical instrument and detector.

All corrective actions will be documented in the field and in laboratory notebooks. If necessary, the DQOs will be revised to meet personnel, equipment, or analytical method capabilities.

5.5 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports will be generated by both the SBC onsite laboratory and offsite contract laboratory to document the analytical results and organizational performance. These reports will contain, at a minimum, system or performance audits; required corrective actions implemented; assessment of the generated data precision, accuracy, and comparability; and resolution of previously reported problems.

6 RECORDKEEPING

Each laboratory will maintain a records system including documentation of all samples received and analyzed, analyses conducted, preparations for analysis, QC challenges, maintenance of laboratory equipment, and reports prepared. All information will become part of the operating record and will be kept in a secured location through closure of the MMD-1 system and preparation and submittal of the final report and conclusion of the MMD-1 test.

Additional records to be retained by the MMD-1 SBC Laboratory Manager will include laboratory performance evaluation results for the past year as well as any responses to errors and associated corrective action. Copies of laboratory certifications will be supplied to the MMD-1 SBC Laboratory Manager.